

**Determination of the viscosity of water at high pressures: Methodology  
and results**

Presented in partial fulfillment of the requirements for  
graduation with a Bachelor of Science in Geological Sciences  
from The Ohio State University

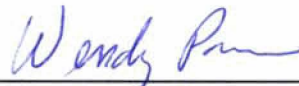
by

Michael C. Veres

The Ohio State University

2007

Approved by



---

Dr. Wendy Panero  
Advisor

### **Acknowledgements**

I would first like to thank Dr. Wendy Panero for providing the idea for this thesis, as well as the use of the lab equipment and assistance during the experiments. I would also like to thank D. M. Reaman for all of his help during the experiments as well as the upkeep of the diamond cell. Additional thanks goes to Veronica McCann for being there to share ideas with. One more thank you goes to my family for helping and supporting me during this thesis.

## **Table of contents**

Abstract .....	1
Introduction .....	1
Methods .....	2
Viscosity of water at high pressures .....	3
Loading the sample .....	4
Results from the high pressure experiments .....	5
Future directions .....	6
Conclusions .....	7
References .....	10

## **Figures**

(1) Stability conditions for hydrous minerals .....	8
(2) Image of the diamond anvil cell and microscope .....	8
(3) Image of a damaged gasket .....	9
(4) Image of a normal gasket with spheres .....	9



## **Abstract**

The viscosity of water can be of major significance to the geology of the mantle wedge. As water dehydrates from a subducting slab, it rises and interacts with the overlying rock of the mantle wedge. The flow rate of water from the slab can lend insight into magma flow below volcanic arcs. In order to measure the viscosity at high pressures (2-5 GPa), a hydrothermal diamond anvil cell (DAC) was used. Brownian motion and viscosity of a fluid can be determined by measuring the displacement of particles in suspension. Prior to using the DAC, analogous experiments were run at room temperature and 1 atmosphere. These analogous experiments resulted in viscosities close to the published values of  $1 \times 10^{-3}$  Pa·s. Problems were encountered with the high pressure experiments. Only low pressures were obtained. The data that were obtained at low pressures indicates that the Brownian motion technique is valid. There are modifications to the experiment that can be done in the future to possibly obtain better results.

## **Introduction**

Volcanic arcs occur across the Earth in connection with subducting oceanic crust. It is thought that dehydration of water from hydrous minerals is the source of the melting. As the slab is subducted, dehydration reactions can occur as shallow as 55 km. Major dehydration then occurs at approximately 120 km depth and is the catalyst for the volcanic arc magmatism (Zhou et al, 2007). Additional dehydration can occur at even greater depths, fueling multiple arcs. The pressure at the dehydration conditions is 2-2.5 GPa with the possibility of dehydration continuing to 6.5 GPa or greater (Kawanoto, 2006; Forneris, 2003).

Kawanoto (2006), Forneris (2003) and Schmidt and Poli (1998) identify the sources for the dehydrated water. Hydrated peridotites, specifically serpentines, chlorite and amphiboles, dehydrate at the above mentioned pressures and depths (see figure 1). These are contained within the basaltic crust and are subducted. The impact of water on the overlying mantle wedge is to reduce the melting temperature enough to melt the mantle peridotites (van Keken, 2003).

The rate of flow for the water released due to dehydration is important. According to Davies (1999), fluid flow is related to intermediate depth earthquakes as well as magmatism. It is proposed that fluid flow from the site of dehydration to the surface could be anywhere between several hundred thousand years to several thousand years. Turner (2001) proposes, based on isotopic ratios, that the rate of fluid flow from the dehydrated slab toward the surface may be as low as 100 years.

The viscosity of water at the pressure conditions present in the mantle wedge could provide a way to see if the rapid flow hypothesis of Turner is possible. There is no existing data for the viscosity of water at these pressure conditions. This was the question that I attempted to answer. If the viscosity at these pressures can be determined, then it may be possible to see if a time of 100 years is even possible.

## **Methods**

There are multiple ways to determine the viscosity of water. One method is the Stokes' flow method. There are two ways to use this method. The first is to determine the time needed for a platinum sphere to roll from one side to another of a container. The other way is to measure the amount of time needed for a particle to settle in a fluid

(Grocholski and Jeanloz, 2005). A third method, the one that I used, is to use Brownian motion to determine the viscosity (Panero et al, 2003).

Brownian motion is small random movements displayed by particles in suspension. The random motions are due to thermal diffusion (Einstein, 1926) and can be seen easily under a petrographic microscope. Brownian motion is only witnessed with very small particles when water is the medium **and** when the particles are suspended – that is, the particles are not stuck to the diamond). The mean square displacement formula can be used to determine viscosity from displacement. The formula is:

$$\langle r^2 \rangle = \left( \frac{4kT}{6\pi\eta a} \right) t$$

Where  $k$  is the Boltzmann constant ( $1.38 \times 10^{-23}$  J/K),  $T$  is the temperature,  $\eta$  is the viscosity,  $a$  is the radius of the particle,  $t$  is the time and  $r$  is the displacement.

### **Proof of concept**

Prior to attempting the experiment at high pressures, an analogous experiment was performed. In order to prove the validity of using a hydrothermal diamond anvil cell for this experiment, the geometry of the cell needed to be duplicated and tested at conditions in which the viscosity of water is well known. Room temperature (approximately 20-25 °C) and 1 atmosphere were chosen as the conditions. The viscosity of water at these conditions is well constrained and is roughly  $1 \times 10^{-3}$  Pa·s. In order to duplicate the geometry of the experiment, a simple set-up was constructed. Water was loaded into 500  $\mu$ m rhenium gasket and was placed between two glass slides. The slides were then pressed together with binder clips. This set-up proved remarkably good at duplicating the geometry of the hydrothermal cell. A viscosity of  $0.5 \times 10^{-3}$  Pa·s was obtained, validating the experimental concept.

### **Viscosity of water at high pressures**

For the high pressure experiment, 3  $\mu\text{m}$  diameter polystyrene spheres (Polybead®) were placed in distilled water. These spheres were large enough to settle, but only if left for a period time much larger than the time needed to run the experiment. The equipment used to obtain the high pressure and temperature conditions was the Bassett-type Hydrothermal Diamond Anvil Cell (Bassett et al, 1993). The diamond anvil cell (DAC) consists of two diamonds with 0.5 mm culets that are placed in an apparatus so that the culets are oriented toward each other. Thermocouples wrap around the diamonds to heat the diamonds and the sample. A rhenium gasket with a 260  $\mu\text{m}$  diameter hole was placed between the culets. The original thickness of the rhenium was about 230 $\mu\text{m}$ . Most of the gaskets used were preindented (compressed) to thickness ranging from 130-170 $\mu\text{m}$ . The entire DAC was placed on a petrographic microscope. A digital camera replaced one of the microscopes eyepieces. During each stage of the experiment, a 40 second video was taken for analysis. Figure 2 shows the DAC connected to the heater prior to one of the experiments.

The DAC does not have a built in pressure sensor. The method devised was to increase the pressure of the sample until the water froze at room temperature. The temperature vs. pressure data from Frank et al (2004) served as a basis to determine the pressure at a given temperature. From there, the temperature would be increased until the sample melted. A video would be taken at this point and then the pressure would be increased.

After the experiment, the particle motion was analyzed from the videos. Using a MATLAB program, the coordinate positions of the particles are automatically calculated.



From this, the displacement, per second, can be obtained and then the viscosity can be determined from the mean square displacement.

### **Loading the sample**

Loading the water into the gasket and then getting the gasket secured within the DAC was challenging. Water's surface tension made it difficult to even get the water into the gasket. The bottom half of the DAC would be placed under a low power microscope, aligning the center of the diamond with a crosshair. From there, the procedure that was used was to use a microsyringe to place a drop of water on one side of the gasket. Then another drop would be placed on the opposite side of the gasket. One side would usually drain through to the other. Once a flow was established through the gasket, another droplet was placed on the bottom diamond. Using the crosshairs as a guide, the gasket was then placed on the bottom diamond, with the side with most of the water up. The top of the DAC was then placed on the posts of the bottom half of the DAC, but the diamonds were not pushed together. The three screws of the DAC were then placed into their holes. One additional drop was then placed on the culet of the *top* diamond. Immediately after placing the drop, the top half of the DAC was lowered all the way down. The screws would have to be tightened quickly (and carefully) to seal the gap between the gasket and the diamonds. If done quickly, the gasket is sealed as the water is still running through it, effectively sealing the water in the gasket. A usable sample was loaded with approximately 50% of the attempts. Care must be taken with the amount of water used. If excessive water is placed on the cement that holds the diamonds to the cell, then the cement can liquefy and the diamond will have to be re-cemented. One method that was used to seal the cement from water was to use an

extremely thin layer (to the point of transparency) of Silly Putty ® to the bottom diamond. Water is unable to soak through and the cement is protected from the water.

### **Results from the high pressure experiments**

There were issues that arose during the experiment. After several attempts, the gasket would deform to the point that the experiment had to be stopped before the water froze. Figure 3 shows the gasket from one of the experiments. The gasket became angular enough that there was a risk of equipment failure if the pressure was increased.

There are some minor errors in the displacements calculated from the video. The MATLAB program that was used required that the video be in the AVI format. During the conversion to the AVI format, some resolution is lost and the finer motions of the particles could fall below this resolution.

The resolution issues were not critical, and probably didn't impact the results. Additional experiments would allow for the error to be reduced and a more precise value for viscosity. The use of Brownian motion to determine the viscosity does appear promising. The data that were obtained fits with existing data. Values in the order of  $10^{-2}$  Pa·s were found at 40 °C and low pressure with viscosity decreasing with increasing temperature.

There was also a problem in that the water never froze, or at least never appeared frozen. There were instances in which the sample appeared to freeze, but began moving again with no change in the equipment. Sometimes, the sample wouldn't freeze at all and the experiment would have to be ended due to the fear of gasket failure. Since the sample did not freeze, the pressures were unknown.

The lack of freezing could have been due to a number of issues. One possibility is that the water had been contaminated with dissolved spheres. The size of the spheres was  $3\mu\text{m} \pm 0.17\mu\text{m}$ . As it can be seen in figure 4, some of the spheres are twice the size of others. This may have been due to dissolution of some of the spheres. Dissolving polystyrene into the water could change the pressures needed to freeze the water. Putting a different material in suspension could check this hypothesis.

The thickness of the gasket proved to be important. As a gasket was drilled, the actual thickness was measured. There appeared to be a minimum thickness for the gasket of about  $170\mu\text{m}$ . If the gasket was thinner than this, there would not be enough space between the culets when pressure was applied. At  $130\mu\text{m}$ , the spheres became adhered to either diamond and there would not be any floating between the diamonds. There was a narrow region between the culets at a gasket thickness of  $170\mu\text{m}$ . A gasket thicker than  $170\mu\text{m}$  would be better. Precompressing the gasket is also important. Uncompressed gaskets deformed rapidly when placed under low pressures.

### **Future directions**

There are two obvious directions that this experiment could be continued. The first was mentioned previously. Use Brownian motion to determine viscosity, but use a different material in place of the polystyrene spheres. The material used could be quartz grains on the scale of a few microns. Another option would be to use the sliding sphere method used by Grocholski and Jeanloz (2005). A platinum sphere would be placed at one side of the DAC and then the microscope is then tilted at a known angle. Viscosity

can be determined by the time needed for the sphere to move from one side to the other. Both of these methods are still dependent on the sample freezing. Another option is to insert a ruby chip into the DAC and measure the internal pressure of the cell from that. In this situation, the experiment would not be restricted to the vicinity of the freezing curve of water.

## **Conclusions**

The purpose of the experiment was to determine the viscosity of water at pressures of at least several GPa, higher than any previous experiments. While the experiment didn't meet with much success, there was still some information that was gained from it. There is a minimum thickness for the gasket of at least 170 $\mu\text{m}$ . There was also an effective procedure developed for loading the sample into the DAC. This method will not work for all liquids, but for water, it works well. The use of quartz grains and Brownian motion or a sliding platinum sphere will allow for the continuation of this project.

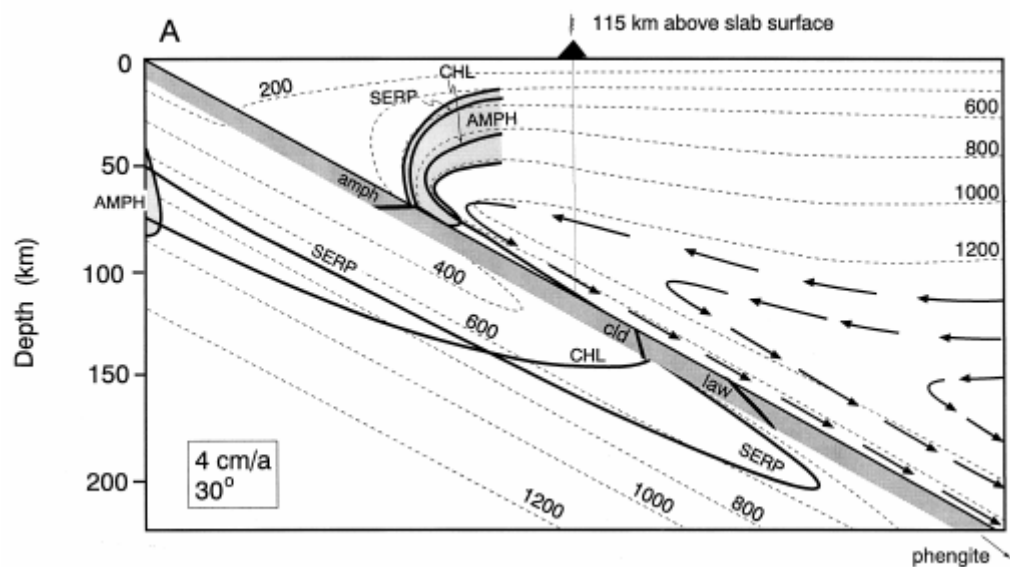


FIGURE 1: Stability pressure and temperature conditions for hydrous minerals in a subducting slab (from Schmidt and Poli, 1998). SERP: Serpentine, AMPH: Amphibole, CHL: Chlorite.

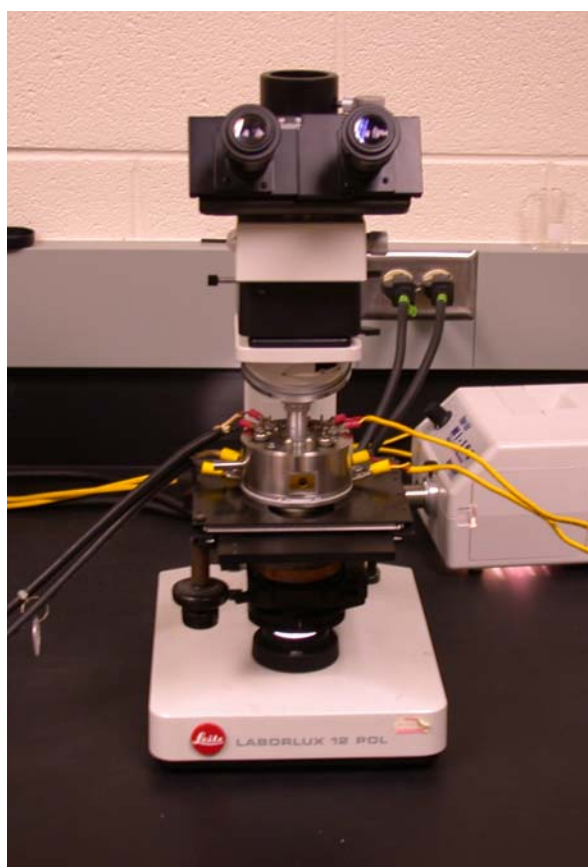


FIGURE 2: Image of the connected DAC prior to one of the experiments.

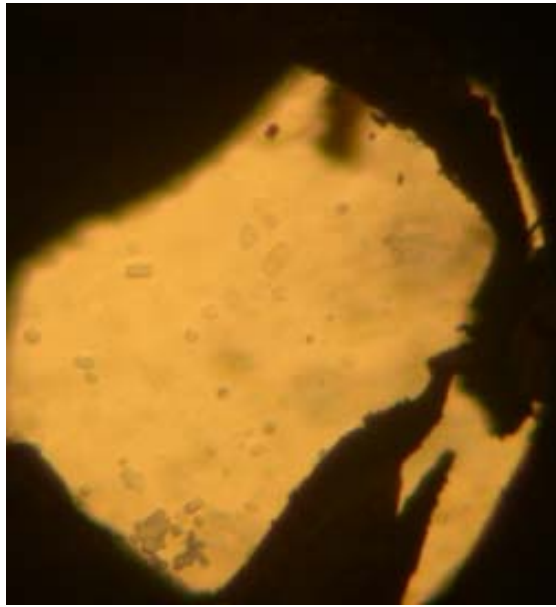


FIGURE 3: Image of a damaged gasket. This image was taken prior to stopping the experiment due to potential gasket failure.

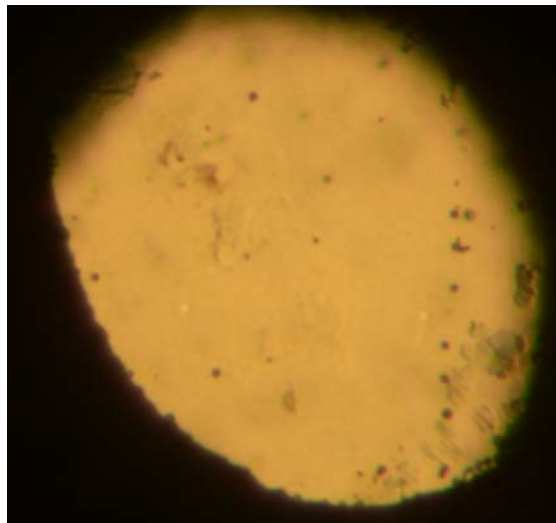


FIGURE 4: Image of a gasket with spheres. Diameter of the gasket is approximately  $260\mu\text{m}$ . Notice the differing sizes of the spheres.

- Bassett, W. A., Shen, A. H. and Bucknum, M., A new diamond anvil cell for hydrothermal studies to 2.5 GPa and from -190 to 1200 °C, Review of Scientific Instruments, v.64, p.2340-2345, 1993.
- Davies, J. Huw, The role of hydraulic fractures and intermediate-depth earthquakes in generating subduction-zone magmatism, Nature, v.398, 11 March, p.142-145, 1999.
- Einstein, Albert, Investigations of the theory of the Brownian Movement, Dover Publications, 119 p., 1926.
- Forneris, Juliette F. and Holloway, John R., Phase equilibria in subducting basaltic crust: implications for H<sub>2</sub>O release from the slab, Earth and Planetary Science Letters, v.214, p.187-201, 2003.
- Grocholski, Brent and Jeanloz, Raymond, High-pressure and -temperature viscosity measurements of methanol and 4:1 methanol:ethanol solution, Journal of Chemical Physics, v.123, 6 p.
- Kawamoto, Tatsuhiko, Hydrous phases and water transport in the subducting slab, in Water in nominally anhydrous minerals, Hans Keppler and Joseph Smyth eds., p.273-289, 2006.
- Panero, W. R., Davis, M. K., Boyd, O. and Stixrude, L. P., Viscosity of silica-rich water at high pressure and temperature, Fall Meeting of the American Geophysical Union, San Francisco, California, December 2003.
- Schmidt, Max W. and Poli, Stefano, Experimentally based water budgets for dehydrating slabs and consequences for arc magma generation, Earth and Planetary Science Letters, v.163, p. 361-379, 1998.
- Frank, Mark R., Fei, Yingwei and Hu, Jingzhu, Constraining the equation of state of fluid H<sub>2</sub>O to 80 GPa using the melting curve, bulk modulus, and thermal expansivity of Ice VII, Geochemica et Cosmochimica Acta, v.68, p.2781-2790.
- Turner, Simon, Ultrafast source-to-surface movement of melt at island arcs from <sup>226</sup>Ra-<sup>230</sup>Th Systematics, Science, v.292, no.5520, p.1363-1366, 2001
- van Keken, Peter E., The structure and dynamics of the mantle wedge, Earth and Planetary Science Letters, v.215, p.323-338, 2003.
- Zhou, Dapeng, Maruyama, Shinegnori and Omori, Soichi, Mantle dynamics of Western Pacific and East Asia: Insight from seismic tomography and mineral physics, Gondwana Research, v.11 no.1-2, p.120-131, 2007.